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Raman spectroscopy during indentation measurements

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A transparent indenter objective described elsewhere [1] allows to observe in real time surface beneath the tip during the measurement placement and monitor its deformation during an indentation. The indenter is diamond-made cylinder faced at the both ends in the shape of the Berkovich pyramid in the way that the top-end pyramid is rotated vs bottom-end pyramid by 60 degrees. The shape allows the parallel beam of light passing through an indenter's face to remain parallel and maintain direction, having only a linear shift vs its original line of propagation. Thus an image observed directly through the objective consists of the three sectors, which form a connected image if the linear translation is applied.

The invention not only saves the time for the movement between indenter and optical objective, but also allows to conduct the spectroscopic measurements during an indentation. An example of phase transformations occurring during the loading and unloading of silicon is given in [2]. In this work we made a measurements of DLC covered silicon specimen. The specimen was loaded up to 40 N and then unloaded back to 30 N after which Raman surface mapping was performed. Corresponding spectra shows a DLC, diamond and Si different phases peaks. Particular peak position depends on the local pressure applied, so one can construct a map for example for the pressure distribution in the DLC coating, which is shown on the Figure 1.

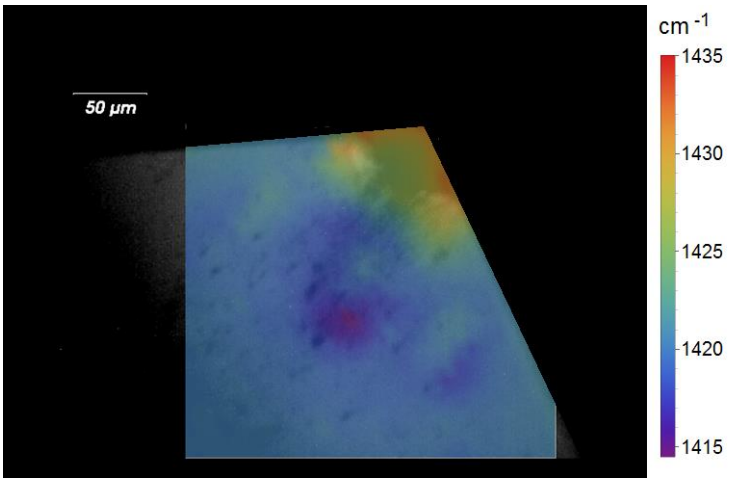

Figure 1. DLC Raman peak position map, 30 N applied.

The map is given for the one out of the three visible sectors and shows that the pressure is bigger within the contact area, particularly below the indenter's edges. One can also observe a dark stains out of the area of indentation, which can be ascribed to the fact that the DLC coatings delaminates from the Si surface, which tends to protrude out of its original position as the radial cracks develops.

The obtained spectra can also be used to create a map for the Si-I/DLC or Si-II/Si-I peak intensity ratio. Conducted experiment shows a possibility to perform a local simultaneous mechanical and spectrum analysis, which can be particular useful for the a sophisticated heterogeneous structures which the phases that undergoes phase transformation under pressure.

1. I.I. Maslenikov, V.N. Reshetov, A.S. Useinov, et al., *Instruments Exp. Tech.*, **61** (5), 719 (2018).
2. I.I. Maslenikov, V.N. Reshetov, A.S. Useinov, *Mater. Trans.* **60** (8), 1433 (2019).